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MTL TR 90-3

PERFORMANCE OF Y-TZP MATERIALS BETWEEN 800°C AND 1200°C

JEFFREY J. SWAB

January 1990

Prepared under
Interagency Agreement DE-AI05-840R21411

by
U.S. ARMY MATERIALS TECHNOLOGY LABORATORY
WATERTOWN, MASSACHUSETTS 02172-0001

Prepared for

U.S. DEPARTMENT OF ENERGY

Assistant Secretary of Conservation and Renewable Energy,
Office of Transportation Systems,
Advanced Materials Development Program

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REPORT DOCUMENTATION PAGE

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1. REPORT NUMBER: MTL TR 90-3	2. GOVT ACCESSION NO.
3. RECIPIENT'S CATALOG NUMBER	
4. TITLE (and Subtitle): PERFORMANCE OF Y-TZP MATERIALS BETWEEN 800°C and 1200°C	5. TYPE OF REPORT & PERIOD COVERED: Final Report
	6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s): Jeffrey J. Swab	8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS: U.S. Army Materials Technology Laboratory, Watertown, Massachusetts 02172-0001, SLCMT-EMC	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: D/A Project: 1L162105.AH84; AMCMS Code: 612105.H840011
11. CONTROLLING OFFICE NAME AND ADDRESS: U.S. Department of Energy, Office of Transportation Systems, Washington, DC 20545	12. REPORT DATE: January 1990
	13. NUMBER OF PAGES: 11
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)	15. SECURITY CLASS. (of this report): Unclassified
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

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17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

Research sponsored by the U.S. Department of Energy, Assistant Secretary for Conservation and Renewable Energy, Office of Transportation Systems, as part of the Ceramic Technology for Advanced Heat Engines Project of the Advanced Materials Development Program, under Contract DE-AI05-840R21411 with Martin Marietta Energy Systems, Inc.

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Zirconia (Y-TZP)
High temperature materials
Strength (flexure)
Ceramic materials
Microstructures

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

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ABSTRACT

The performance of seven yttria-tetragonal zirconia polycrystal materials in the 800°C to 1200°C temperature range was examined using stepped-temperature stress-rupture tests. The strength in this temperature range was significantly lower than the room temperature strength. This is due to a decrease in the chemical driving force for the tetragonal-to-monoclinic transformation. As a result, these materials are not suitable for high temperature structural applications. At these temperatures the strength is controlled by microstructural parameters such as grain size, grain size distribution, and porosity.

FOREWORD

The work described herein is part of the Characterization of Transformation-Toughened Ceramics Program which is a subtask of the Department of Energy (DOE) sponsored, Oak Ridge National Laboratory (ORNL) monitored, Ceramic Technology for Advanced Heat Engines Project (Interagency Agreement No. DE-AI05-84OR21411). The purpose of this subtask is to examine commercial and experimental transformation-toughened ceramics for potential application in advanced heat engines.

INTRODUCTION

Tetragonal zirconia polycrystal (TZP) materials have been considered for structural applications because of an unusual combination of high strength and toughness at room temperature. In addition, the thermal expansion is closely matched to cast iron and some other steels, thus reducing potential thermal expansion mismatch problems in ceramic/metal attachments. The excellent mechanical properties are the result of a "martensitic" type transformation where metastable tetragonal grains transform to the stable monoclinic grains under an applied stress.[1-3] Although TZP materials have this unusual combination of properties, little has been done to examine the time-dependent behavior of the properties at elevated temperatures.[4,5] Most of the elevated temperature work has concentrated on compressive or tensile creep behavior at $\geq 1200^{o}C$ relating to the phenomenon of superplasticity.[6-15] This lack of data generation can be attributed to the well defined polymorphism of zirconia.[16] The monoclinic phase is stable at room temperature while the tetragonal phase becomes the stable phase above $\sim 950^{o}C$. Thus, as the temperature increases so does the stability of the tetragonal phase, with a corresponding decrease in the chemical driving force for the $t \rightarrow m$ transformation, resulting in a significant decrease in strength and toughness at temperature. Because of this inherent problem, the use of monolithic TZP materials for high temperature ($\geq 1000^{o}C$) structural applications (i.e., heat engines) have been severely limited. This report summarizes a study to examine the time-dependent strength of several TZP materials at temperatures between $800^{o}C$ and $1200^{o}C$.

EXPERIMENTAL PROCEDURE

Bend bars were machined from the seven different yttria-tetragonal zirconia polycrystal (Y-TZP) materials listed in Table 1. The first six materials were machined to the following dimensions: 3 mm x 4 mm x 50 mm. Due to material limitations, the seventh material (AC) was machined into smaller bend bars, 1.5 mm x 2 mm x 25 mm. In both cases, the bars were carefully ground by a surface grinder such that the surface striations are parallel to the long axis. All four edges were chamfered to $\sim 45^{o}$. The bars were machined according to the details specified in Reference 17.

Table 1. EVALUATED MATERIALS

	Code	Manufacturer	Material	Process	Mole % Y_2O_3
Japanese					
	KY	Kyocera	Z-201	Sintered	2.8
	TOSH	Toshiba	TASZIC	Sintered	2-3
	HIT	Hitachi	1985	Hot Pressed (?)	2.0
	NGK	NGK Locke	Z-191	Sintered	3.0
	KS	Koransha	1986	Sintered	3.0
	KH	Koransha	1986	HIPed	3.0
Domestic					
	AC	AC Sparkplug	TZP-110	Sintered	2.6

Stepped-temperature stress-rupture (STSR) testing was used to analyze the time-dependent strength of these Y-TZPs between 800°C and 1200°C. STSR testing was done following the procedure outlined by Quinn and Katz.[18] This type of test allows for rapid screening of the materials' stress-rupture behavior over a wide range of temperatures while using a small number of specimens. This procedure involved loading a bar onto a four-point bend fixture that is in a furnace and heating the furnace to 800°C in 2 hours, in air, with no applied stress to the bar. Upon reaching the temperature, a predetermined stress is applied and the bar allowed to soak for up to 24 hours. If the bar survives this step, then the furnace is heated to 900°C (in ~10 min) while under the same applied stress and again the bar is allowed to soak for 24 hours. This cycle is repeated for 1000°C, 1100°C, and 1200°C. If the bar fractures or excessive creep occurs, the power to the furnace is automatically shut off by a microswitch. The time of fracture is denoted on the STSR plot using an arrow, with the applied stress that caused fracture above the arrow. The symbols for the STSR plots are: (←) failure occurred upon application of the stress at 800°C; (→) survived full test cycle through 1200°C; and (↓) denotes time of failure between application of the stress, but before the full cycle is complete.

RESULTS AND DISCUSSION

Material Characterization

Table 2 lists the base line room temperature properties of each TZP material.

Table 2. BASE LINE ROOM TEMPERATURE PROPERTIES

	HIT	NGK	KH	KS	KY	AC	TOSH
Density (g/cc)	6.0	5.9	6.1	6.0	5.9	5.8	5.9
MOE (GPa)	213	208	214	210	201	204	200
MOR (MPa)	1169	873	1261	640	745	753	633
K_{Ic} (MPa*m$^{1/2}$)	4.6	7.4	5.0	5.6	7.0	5.6	8.0
Average Grain Size (μm)	0.4	0.2	0.4	0.5	0.7	0.8	0.5

Further details on the techniques used to determine the base line properties and the microstructure of each TZP can be found in Reference 19.

Stepped-Temperature Stress-Rupture

It can clearly be seen from Figures 1 through 7 that none of these TZPs will be able to handle the combination of high temperature (>982°C) and stress (>800 MPa) which are desired of materials for advanced heat engine applications.[20] These STSR results are in excellent agreement with the STSR results for two other yttria partially stabilized zirconias which were examined in Reference 4.

For all TZPs, the strength at any of the temperatures is significantly below the room temperature strength and this difference grows as the temperature increases. This strength loss is due to the previously mentioned decrease in the chemical driving force for the t→m transformation as the temperature increases.

Therefore, as this driving force decreases, the strength at temperature will become increasingly dependent on the microstructure. Since previous studies[19,21] have shown that there is little or no grain boundary phases present in any of these TZPs, the important microstructural parameters will be grain size, grain size distribution, and size and amount of strength limiting flaws.

Table 2 shows that HIT, NGK, and KH have the finest grain size and it has been shown that they have the narrowest grain size distribution[21] and the smallest strength limiting flaws.[19] Thus, it is expected that they will perform better than the remaining TZPs. This is true for HIT (Figure 1) and NGK (Figure 2) but not KH (Figure 3). The performance of HIT is slightly better than NGK even though its grain size is twice as large. This is due to the narrow grain size distribution, which shows that over 90% of the grains are between 0.2 and 0.5 μm and the remainder are no larger than 0.7 μm. NGK also has a narrow distribution but a small percentage of the grains are in excess of 1 μm. The other contributing factor is that HIT is hot pressed while NGK is sintered. Hot pressing helps to maintain this fine grain size and narrow distribution by inhibiting grain growth, and it reduces the amount and size of porosity related flaws. Sintering does not reduce porosity as much as hot pressing, and grain growth is common to this technique.

The KH has a grain size distribution similar to NGK but its poor performance is a result of oxygen deficiency that arises when the TZP is HIPed in an inert atmosphere. The details of this phenomenon are described elsewhere.[5] If the KH is oxidized in air to restore stoichiometry, then the STSR performance in this temperature range becomes similar to NGK.

The performance of the remaining TZPs (Figures 4 through 7) is similar and is a result of their larger grain size, broader grain size distribution, and larger amount of bulk porosity. In addition, the room temperature strength of these TZPs is limited by large porous regions (50 to 100 μm).[19] These porous regions are 3 to 4 times larger than encountered in the other TZPs.

A final point is that at the higher temperatures (1100°C and 1200°C) there is evidence of creep in all TZPs. Because of the fine grain size of each TZP, this is probably the occurrence of Nabarro-Herring or Coble creep since these types of creep dominate in fine-grained polycrystalline materials. An analysis to determine if these mechanisms are present is beyond the realm of this study and is probably inconsequential since the performance of these materials is well below what is required for application in advanced heat engines.

CONCLUSIONS

Stepped-temperature stress-rupture testing of seven Y-TZP materials in the 800°C to 1200°C range shows that these materials are not suitable for use as a high temperature structural ceramic. In this temperature range, the strength is significantly lower than at room temperature due to a decrease in the chemical driving force for the t→m transformation. At these temperatures, the strength is controlled by microstructural parameters such as grain size, grain size distribution, and porosity.

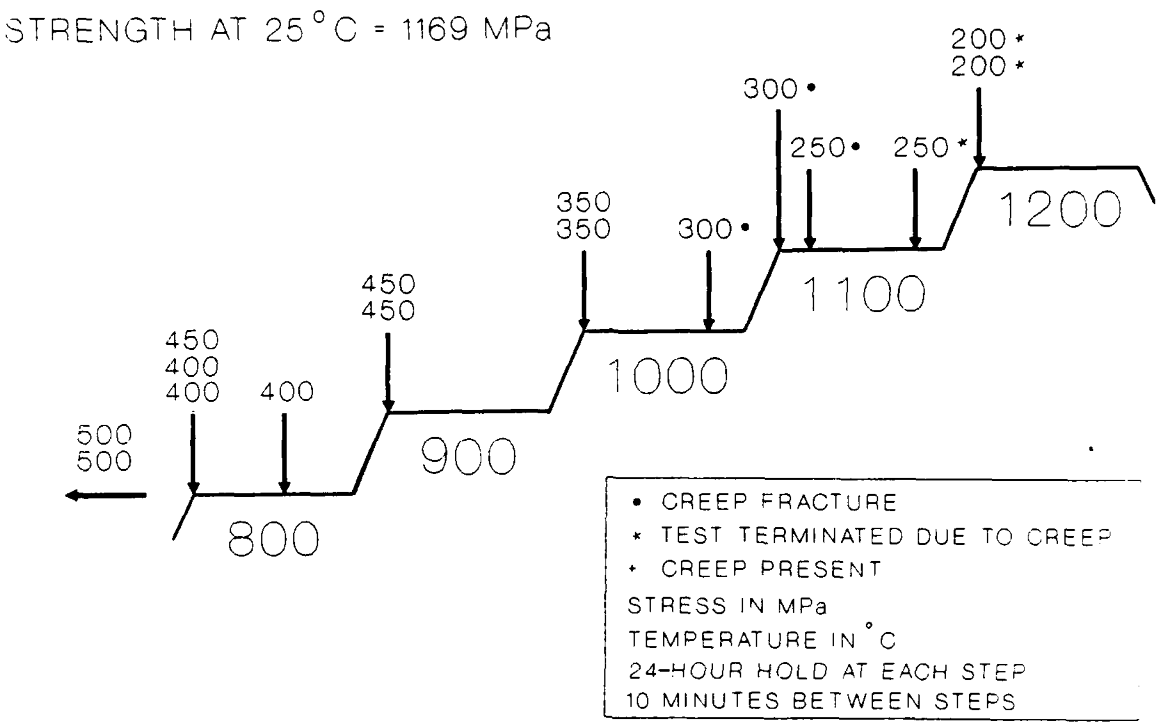

Figure 1. Stepped-temperature stress-rupture results for Hitachi 1985 (HIT).

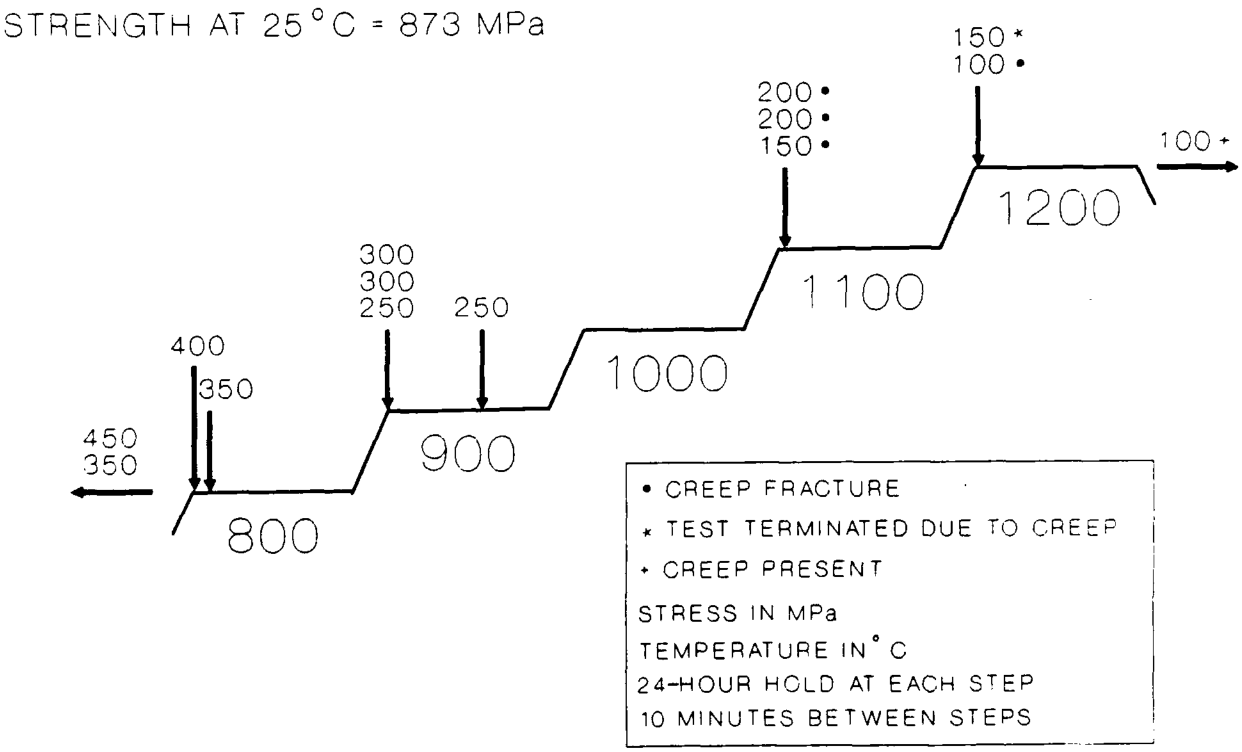

Figure 2. Stepped-temperature stress-rupture results for NGK-Locke Z-191 (NGK).

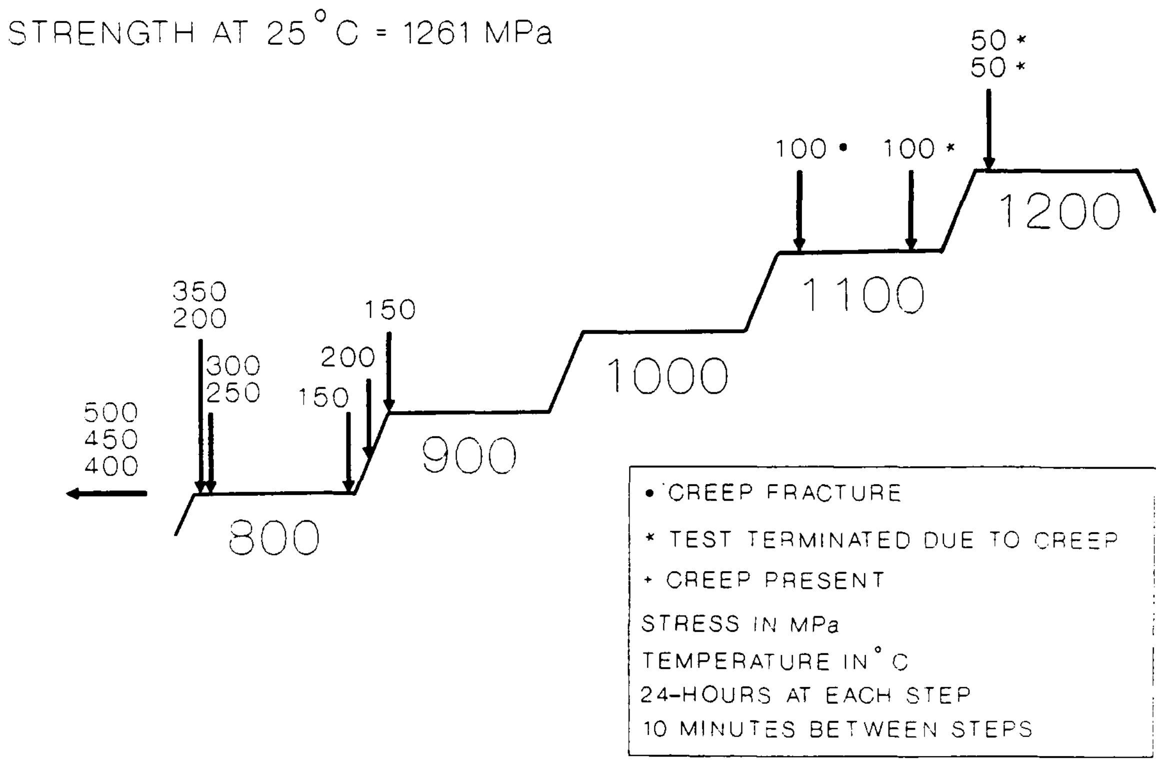

Figure 3. Stepped-temperature stress-rupture results for Koransha HIPed (KH).

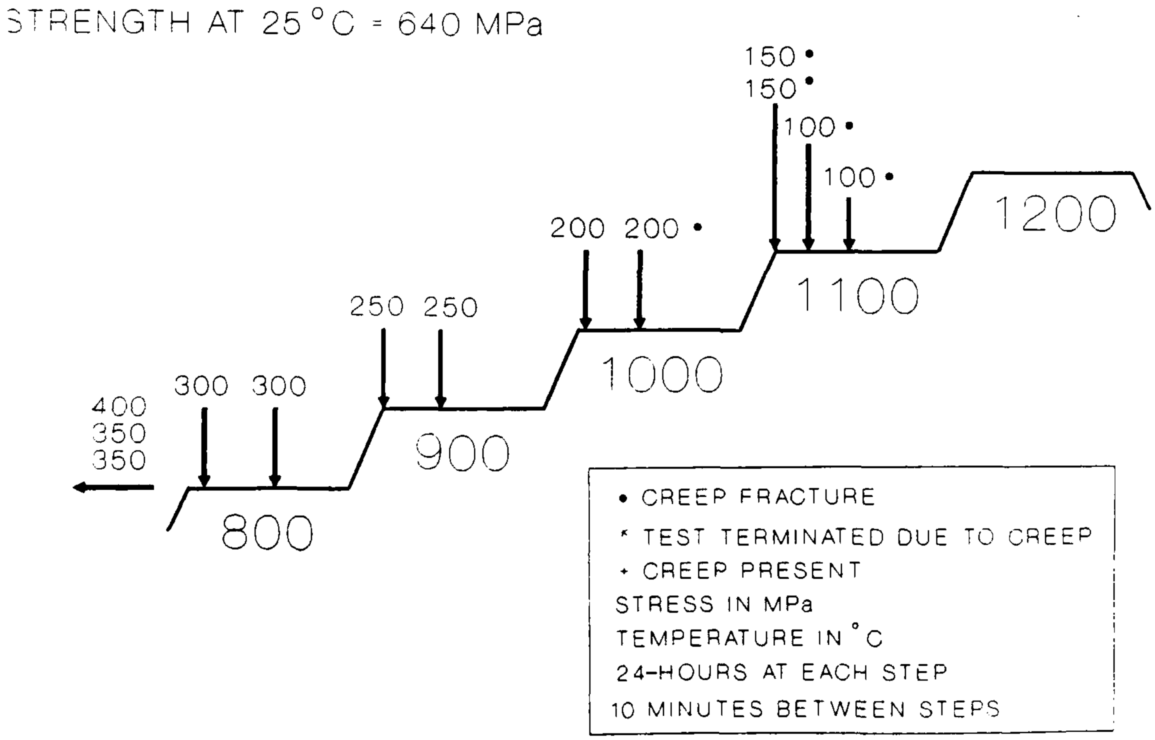

Figure 4. Stepped-temperature stress-rupture results for Koransha Sintered (KS).

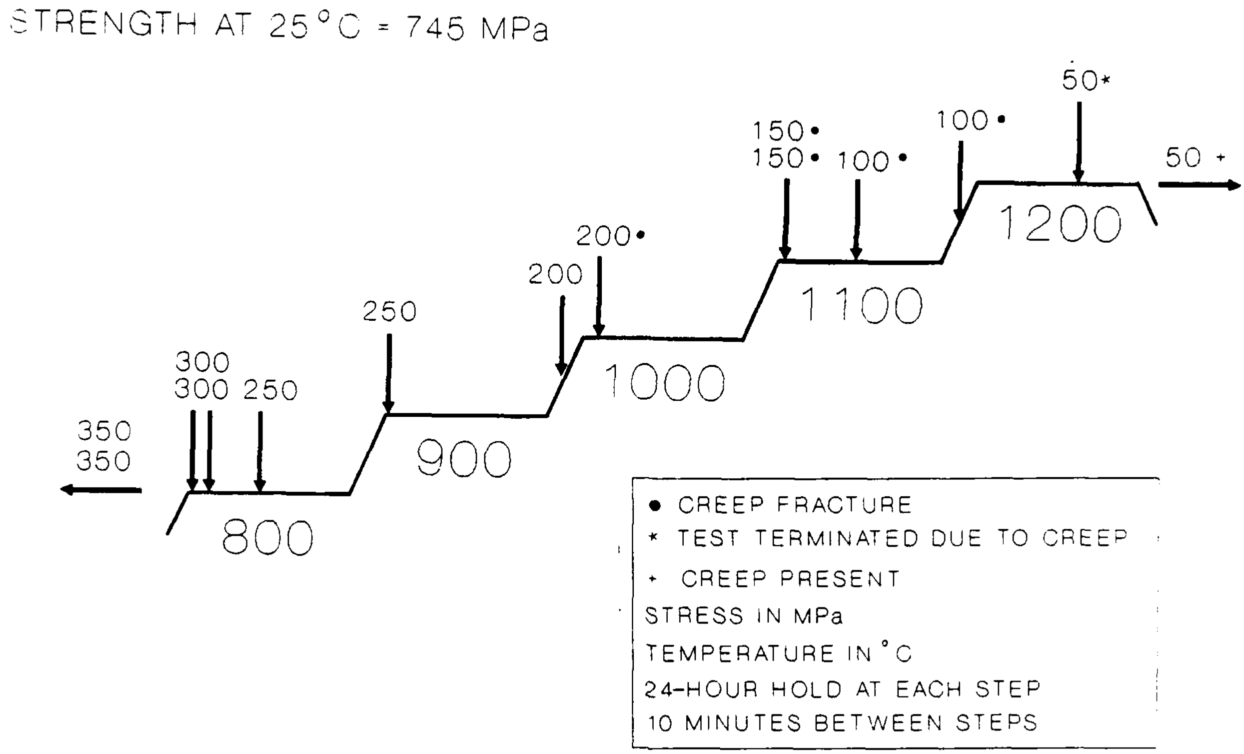

Figure 5. Stepped-temperature stress-rupture results for Kyocera Z-201 (KY).

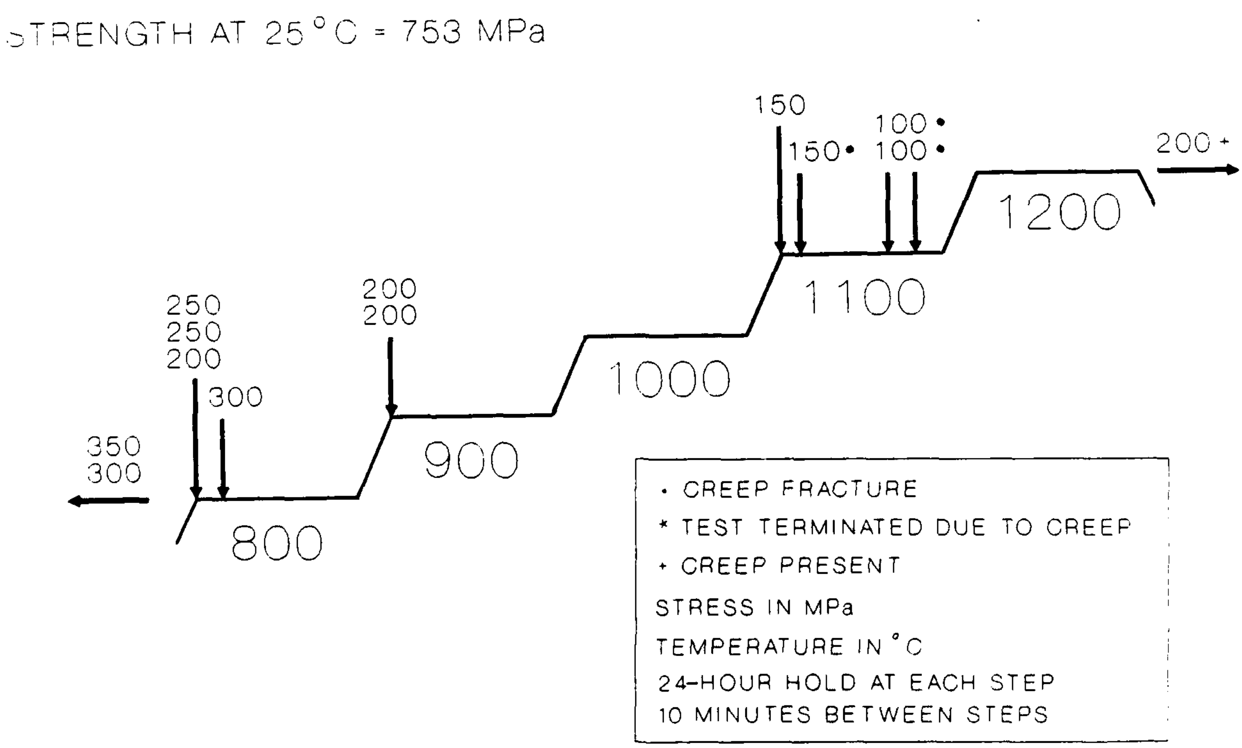

Figure 6. Stepped-temperature stress-rupture results for AC Sparkplug TZP-1-10 (AC).

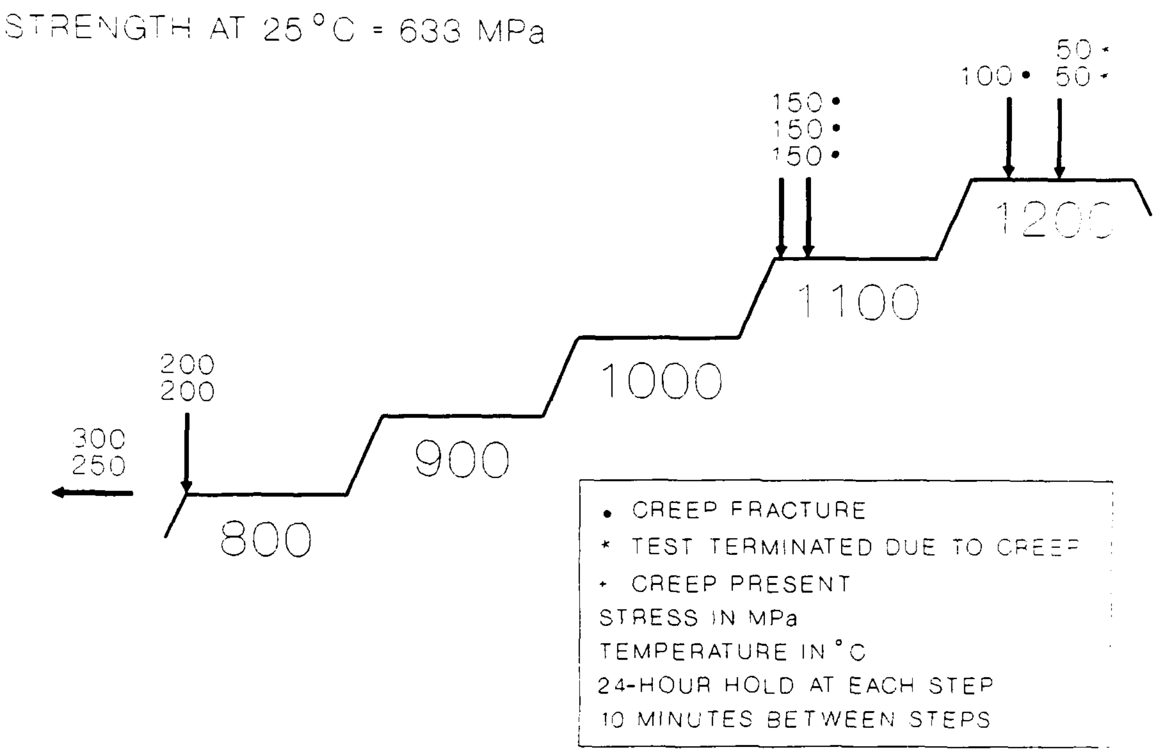

Figure 7. Stepped-temperature stress-rupture results for Toshiba TASZIC (TOSH).

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Key Words

Zirconia (Y-TZP)
High temperature materials
Strength (flexure)

The performance of seven yttria-tetragonal zirconia polycrystal materials in the 800°C to 1200°C temperature range was examined using stepped-temperature stress-rupture tests. The strength in this temperature range was significantly lower than the room temperature strength. This is due to a decrease in the chemical driving force for the tetragonal-to-monoclinic transformation. As a result, these materials are not suitable for high temperature structural applications. At these temperatures the strength is controlled by microstructural parameters such as grain size, grain size distribution, and porosity.